Crystal structure of tetraaqua diacetato-κ^2O,O'-4,4',6,6'-tetramethyl-2,2'-bipyrimidine-$\kappa^4N,N':N'',N'''$ dicobalt(II) diacetate dihydrate, $C_{10}H_{19}CoN_2O_7$

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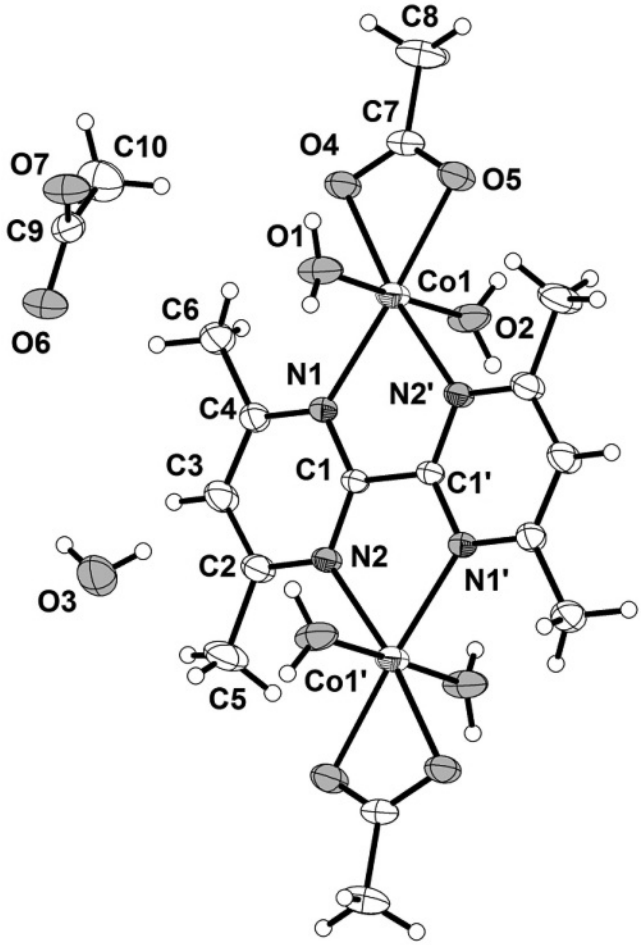

Abstract

$C_{10}H_{19}CoN_2O_7$, triclinic, $P\bar{1}$ (no. 2), a = 8.622(2) Å, b = 8.724(2) Å, c = 10.600(2) Å, α = 78.507(2)°, β = 88.045(2)°, γ = 71.118(2)°, V = 738.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.0248, $wR_{ref}(F^2)$ = 0.0667, T = 296 K.

Source of material

A mixture of $Co(OAc)_2{\cdot}4H_2O$ (0.100 g, 0.4 mmol) and *tmbpm* (4,4',6,6'-tetramethyl-2,2'-bipyrimidine, 0.043 g, 0.2 mmol) in H_2O (12 mL) was stirred for 30 min at room temperature. Then the solution was transferred and sealed in a 20 mL Teflon-lined stainless vessel which was heated at 140 °C for three days. The solution cooled to room temperature at a rate of 2.3 °C/h. Red block-shaped crystals of the title compound were obtained.

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Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times U_{eq}(aryl C), or $U_{iso}(H) = 1.5$ times U_{eq}(methyl C or water O) and included in the final refinement by using geometrical restraints, with C–H = 0.93 Å (aryl) or C–H = 0.96 Å (methyl), and O–H = 0.84 Å.

Table 1. Data collection and handling.

Crystal:	red blocks, size 0.17×0.19×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.92 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5677, 2729
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2563
$N(param)_{refined}$:	185
Programs:	SHELX [8]

Discussion

2,2'-bipyrimidine (*bpm*) has been extensively used in the development of metal complexes [1, 2]. As a versatile ligand, *bpm* is able to coordinate metals ions in a bidentate or bis-bidentate bridging mode, leading to mono or polynuclear complexes [3]. A large number of uncommon magnetic systems have been reported as a consequence of the growing research on the magnetic properties of *bpm*-bridged complexes [4, 5]. Some bridging ligands are able to construct metal complexes with interesting properties for potential application. We want to investigate the factors influencing the coordination mode of the ligands and which factors control the final structure of the complexes. We select 4,4',6,6'-tetramethyl-2,2'-bipyrimidine (*tmbpm*), the derivatives of *bpm*, to design the expect complexes. The structure of the title compound consists of discrete $[Co_2(tmbpm)(CH_3COO)_2(H_2O)_4]^{2+}$ units, CH_3COO^- anions and solvent water molecules (Fig.). The two Co(II) atoms are bridged by a *tmbpm* ligand. Each Co^{2+} is in a slightly distorted octahedral environment: two nitrogen atoms from the *tmbpm* (2.173 Å for Co1–N1 and 2.168 Å for Co1–N2) and two oxygen atoms from the acetate moiety (2.170 Å for Co1–O4 and 2.135 Å for Co1–O5) build the equatorial plane, while the axial position are occupied by two water oxygen atoms (2.035 Å for Co1–O1 and 2.054 Å for Co1–O2). The Co–N bond lengths are similar to those in the cobalt complexes of 2,2'-bipyrimidine (*bpm*) [6, 7]. The Co(*tmbpm*)Co unit is planar with the largest deviation of 0.107 Å for C5, and the Co–Co distance is 5.795 Å. The hydrogen bonding interactions play an important role in stabilizing the title crystal structure. The $[Co_2(tmbpm)(CH_3COO)_2(H_2O)_4]^{2+}$ units, uncoordinated CH_3COO^- anions and free water molecules are linked by intermolecular hydrogen bonds to form a three-dimensional network.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1W)	2*i*	0.8913	0.5111	0.6295	0.069
H(2W)	2*i*	0.8378	0.6346	0.5248	0.069
H(4W)	2*i*	0.4372	0.9554	0.7976	0.072
H(3W)	2*i*	0.4908	0.8347	0.9060	0.072
H(5W)	2*i*	0.5085	0.8045	0.1456	0.091
H(6W)	2*i*	0.4318	0.7000	0.1179	0.091
H(3)	2*i*	0.1425	0.7745	0.4267	0.048
H(5A)	2*i*	0.0443	1.1835	0.2735	0.091
H(5B)	2*i*	–0.0015	1.0235	0.2740	0.091
H(5C)	2*i*	0.1347	1.0618	0.1837	0.091
H(6A)	2*i*	0.4609	0.5208	0.6510	0.067
H(6B)	2*i*	0.3159	0.5143	0.5692	0.067
H(6C)	2*i*	0.2794	0.5939	0.6917	0.067
H(8A)	2*i*	0.8859	0.4036	1.0729	0.077
H(8B)	2*i*	0.8065	0.2843	1.0282	0.077
H(8C)	2*i*	0.6999	0.4261	1.0956	0.077
H(10A)	2*i*	0.8686	0.7152	0.0940	0.089
H(10B)	2*i*	0.9907	0.7904	0.1450	0.089
H(10C)	2*i*	0.8148	0.9052	0.0932	0.089

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co(1)	2*i*	0.64451(3)	0.72535(3)	0.71044(2)	0.0278(1)	0.0234(1)	0.0216(1)	–0.0052(1)	–0.00400(9)	0.00164(9)
O(1)	2*i*	0.8131(2)	0.5957(2)	0.5989(1)	0.0472(8)	0.0396(8)	0.0308(7)	0.0082(6)	0.0038(6)	0.0018(6)
O(2)	2*i*	0.4799(2)	0.8554(2)	0.8268(1)	0.063(1)	0.0341(7)	0.0275(7)	0.0052(7)	0.0052(7)	0.0012(6)
O(3)	2*i*	0.4560(2)	0.7854(2)	0.0899(2)	0.092(1)	0.062(1)	0.0376(9)	–0.044(1)	–0.0067(9)	0.0036(8)
O(4)	2*i*	0.6371(2)	0.4986(2)	0.8356(1)	0.0439(8)	0.0363(7)	0.0336(7)	–0.0152(6)	–0.0104(6)	0.0067(6)
O(5)	2*i*	0.8083(2)	0.6214(2)	0.8748(1)	0.0414(8)	0.0432(8)	0.0331(7)	–0.0161(6)	–0.0100(6)	0.0052(6)
O(6)	2*i*	0.9135(2)	0.6804(2)	0.3629(1)	0.0361(8)	0.0493(9)	0.0384(8)	–0.0011(7)	–0.0016(6)	0.0026(7)
O(7)	2*i*	0.6620(2)	0.8297(2)	0.2875(1)	0.0392(8)	0.0454(8)	0.0417(8)	0.0025(7)	0.0030(7)	0.0041(7)
N(1)	2*i*	0.4514(2)	0.8137(2)	0.5615(1)	0.0286(7)	0.0240(7)	0.0209(7)	–0.0083(6)	–0.0016(6)	–0.0009(5)
N(2)	2*i*	0.3266(2)	1.0454(2)	0.3936(1)	0.0268(7)	0.0268(7)	0.0239(7)	–0.0083(6)	–0.0044(6)	0.0007(6)
C(1)	2*i*	0.4385(2)	0.9609(2)	0.4876(2)	0.0241(8)	0.0232(8)	0.0189(8)	–0.0056(7)	–0.0018(6)	–0.0008(6)
C(2)	2*i*	0.2133(2)	0.9759(2)	0.3704(2)	0.036(1)	0.037(1)	0.033(1)	–0.0149(8)	–0.0107(8)	0.0033(8)
C(3)	2*i*	0.2193(3)	0.8232(3)	0.4427(2)	0.044(1)	0.043(1)	0.039(1)	–0.026(1)	–0.0133(9)	0.0043(9)
C(4)	2*i*	0.3393(2)	0.7430(2)	0.5386(2)	0.038(1)	0.0306(9)	0.0263(9)	–0.0158(8)	–0.0025(8)	–0.0007(7)
C(5)	2*i*	0.0863(3)	1.0696(3)	0.2661(3)	0.060(2)	0.059(2)	0.061(2)	–0.032(1)	–0.039(1)	0.021(1)
C(6)	2*i*	0.3498(3)	0.5782(3)	0.6199(2)	0.059(1)	0.039(1)	0.038(1)	–0.029(1)	–0.011(1)	0.0076(9)
C(7)	2*i*	0.7440(2)	0.5106(2)	0.9090(2)	0.0304(9)	0.033(1)	0.028(1)	–0.0016(8)	–0.0021(8)	0.0031(8)
C(8)	2*i*	0.7881(3)	0.3958(3)	1.0381(2)	0.045(1)	0.060(1)	0.033(1)	–0.006(1)	–0.0043(9)	0.014(1)
C(9)	2*i*	0.8130(2)	0.7674(2)	0.2730(2)	0.037(1)	0.0288(9)	0.033(1)	–0.0062(8)	0.0015(8)	–0.0041(8)
C(10)	2*i*	0.8777(3)	0.7973(3)	0.1391(2)	0.059(2)	0.070(2)	0.040(1)	–0.011(1)	0.011(1)	–0.010(1)

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